Evaluation of Thermal Control Coatings for Flexible Ceramic Thermal Protection Systems

Demetrius Kourtides, Carol Carroll, Mike Guzinski, Jochen Marschall, Joan Pallix, Jerry Ridge, Dane Smith, and Duoc Tran

July 1997

National Aeronautics and
Space Administration

Evaluation of Thermal Control Coatings for Flexible Ceramic Thermal Protection Systems

Demetrius Kourtides, Carol Carroll, Dane Smith, Ames Research Center, Moffett Field, California

Mike Guzinski, Jochen Marschall, Joan Pallix, Jerry Ridge, and Duoc Tran, Thermosciences Institute, Moffett Field, California

July 1997

National Aeronautics and
Space Administration

Ames Research Center
Moffett Field, California 94035-1000

Evaluation of Thermal Control Coatings for Flexible Ceramic Thermal Protection Systems

DEMETRIUS KOURTIDES, CAROL CARROLL, MIKE GUZINSKI,[*]

JOCHEN MARSCHALL,[*] JOAN PALLIX,[*] JERRY RIDGE,[*] DANE SMITH, AND DUOC TRAN[*]

Ames Research Center

Summary

[*]This report summarizes the evaluation and testing of high emissivity protective coatings applied to flexible insulations for the Reusable Launch Vehicle technology program. Ceramic coatings were evaluated for their thermal properties, durability, and potential for reuse. One of the major goals was to determine the mechanism by which these coated blanket surfaces become brittle and try to modify the coatings to reduce or eliminate embrittlement. Coatings were prepared from colloidal silica with a small percentage of either SiC or SiB_6 as the emissivity agent. These coatings are referred to as gray C-9[1] and protective ceramic coating (PCC), respectively. The colloidal solutions were either brushed or sprayed onto advanced flexible reusable surface insulation blankets. The blankets were instrumented with thermocouples and exposed to reentry heating conditions in the Ames Aeroheating Arc Jet Facility. Posttest samples were then characterized through impact testing, emissivity measurements, chemical analysis, and observation of changes in surface morphology. The results show that both coatings performed well in arc jet tests with backface temperatures slightly lower for the PCC coating than with gray C-9. Impact testing showed that the least extensive surface destruction was experienced on blankets with lower areal density coatings.

Introduction

Advanced Flexible Reusable Surface Insulation (AFRSI) blankets have been used successfully on many space shuttle missions. The shuttle AFRSI blankets are coated with a silica based coating called C-9 which provides protection to the outer mold line (OML) fabric by closing pores in the surface to reduce the flow of hot gas to the interior. Standard shuttle AFRSI is composed of pure silica fabric with a pure silica batting (Q-felt) and is used in applications where the surface temperatures do not exceed 1200–1500°F as on the leeward surface of the orbiter. A new AFRSI blanket is in the development stage at Rockwell International and at Ames Research Center. The OML of this blanket is woven in an angle interlock weave from yarns consisting of Nextel 440 fibers made by 3M.

Nextel 440 ceramic fibers are continuous polycrystalline aluminoborosilicate fibers composed (by weight) of 70% Al_2O_3, 28% SiO_2, and 2% B_2O_3 (ref. 1). The batting material is Saffil which is composed of 97% Al_2O_3 and 3% SiO_2. The high alumina content of the blanket increases the temperature capabilities so that it can be used in applications where surface temperatures reach ~2000°F. A high emittance coating for this new AFRSI blanket will also be required for use in high temperature environments of a convective nature.

Prior to the Reusable Launch Vehicle (RLV) technology program, attempts to produce a higher temperature coating involved attempts to mix colloidal alumina with colloidal silica. This would increase the temperature capabilities of the coating and result in a coating with thermal expansion properties similar to those of the blanket material. Preliminary work done both at Ames and at Rockwell indicated that this process is difficult at best. The mixture of colloidal alumina and silica forms a gel shortly after mixing. The mixture can be stored in a freezer for short periods of time but still gels as soon as it is removed.

Throughout the duration of the Ames-Rockwell cooperative agreement there were two satisfactory coatings that were developed and tested. Both coatings were made by adding a high emissivity agent to a mixture of silica, colloidal silica, and water. The coatings studied in this work are gray C-9 and protective ceramic coating (PCC), which contain the emissivity agents SiC and SiB_6, respectively.

---

[*] Thermosciences Institute, Moffett Field , CA 94035
[1] Note that the gray C-9 referred to throughout this report is used on the shuttle to coat gap fillers and is sometimes referred to as C-10.

The PCC coating was developed at Ames and the gray C-9 coating was formulated by Rockwell International. These high temperature protective coatings have been described previously in detail (refs. 2 and 3). Once the proper formulation for the coatings was established, it was necessary to determine the best method of applying the coating to the AFRSI surface with a minimum weight impact. Brush-on and spray-on application processes were developed, and samples prepared by both methods were tested in the Ames Arc Jet Facility.

This report summarizes the results of arc jet and other tests that were carried out to evaluate the durability and characterize the thermal and structural properties of AFRSI blankets treated with these high emissivity coatings. Pre and post arc jet–tested samples were also analyzed for changes in emissivity, surface chemistry, surface morphology, and resistance to impact in order to understand the factors that contribute to surface embrittlement after multiple reentry cycles.

Results

Arc Jet Testing

The 20MW Aeroheating Facility (AHF), at NASA Ames Research Center, was used to expose the gray C-9 and PCC surface coatings on Nextel 440 AFRSI blankets to multiple simulated reentry cycles as described in tables 1(a) and 1(b). Two application methods were used to apply the different coatings onto the OML of the Nextel 440 AFRSI blanket.

The spray-on method consisted of several passes with a spray gun over the surface until a desired wet weight was obtained. Each pass can only deposit a small amount of coating material due to limitations of the spray gun. In the brush-on method of application, the coating is brushed until complete coverage of the OML has been achieved. The desired amount of coating to apply in one application is calculated to achieve a certain weight per unit area upon drying. The advantage of the brush-on method is that it achieves good penetration of the coating into a given weave of yarns.

The PCC coating was applied by the spray-on method as well as the brush-on method. The spray-on method provided a more controllable application of the PCC coating at the lowest areal density (0.035 lb/ft^2). For these tests, only the brush-on method was used to apply the gray C-9. This application of the gray C-9, which is composed of standard shuttle C-9 coating with a small percentage of SiC or "gray" additive, was done to compare with the standard coated shuttle C-9 per shuttle specification (brush-on).

Blankets were instrumented with thermocouples on the OML and inner mold line (IML) fabrics. The OML thermocouple was attached on the backside of the OML fabric.

After the application of the coatings, 3-1/2in.^{2}, 1 inch thick AFRSI samples were inserted into a 6 1/4 inches diameter, nonablative ceramic model holder. The coated samples were evaluated in the 20MW AHF Arc Jet Facility and the thermal performance of each sample was recorded. These samples were subjected to aeroheating of 2000°F on the coated surface for approximately 9 minutes per cycle at multiple aerothermal cycles. After each of the 9 minute cycles, the maximum back-face temperature of the AFRSI blanket and the elapsed time were recorded. Tables 1(a) and 1(b) summarize the test parameters—duration, temperature, and AHF settings. Figure 1 represents the typical aerothermal cycle of the coated samples in the arc jet facility.

The averages of the thermal performance parameters of the coatings after multiple exposures are shown in figure 2. The bar graph shows the surface temperature read by the infrared, optical pyrometer during model exposure in the arc jet flow stream. Figure 2 also shows the average maximum back-face temperature and the time elapsed from model insertion into the stream to peak back-face temperature. The low areal density PCC sample reached a maximum of about 510°F at the average elapsed time of 1360 seconds. Other higher areal density coated PCC samples yielded similar back-face temperatures, but slightly faster thermal conduction rates, or shorter elapsed times.

Table 1(a). Summary results of arc jet tests on PCC coated blankets

Cum [a] Exp	Run No.	Time In Arc (s)	Max Surface Temp °F	Time to Max BF[b] (s)	Max BF Temp °F	Chamber Pressure PSIA	Stagnation Pressure PSIA	Calc. Heat Flux Rate Btu/ft²s
Sample 1[c]								
1	17	534	2006	1190	499	19	0.630	15.0
2	18	532	2002	1274	489	20	0.658	14.9
3	19	541	2005	1324	480	21	0.685	15.0
4	20	538	2045	1323	522	22	0.713	16.0
5	23	555	2013	1333	519	20	0.658	15.2
6	24	540	2012	1325	515	20	0.658	15.1
	Average	540.1	2013.9	1294.8	504.0	20.3	0.667	15.2
	Std Dev	8.1	15.7	55.6	17.5	1.0	0.029	0.4
Sample 2[d]								
1	11	535	2019	1176	568	18	0.602	15.3
2	12	534	2007	1207	565	17	0.574	15.0
3	13	527	2002	1144	509	17	0.574	14.9
4	14	534	2014	1196	518	17	0.574	15.2
5	15	544	2041	1298	561	19	0.630	15.9
6	16	542	2024	1178	568	17	0.574	15.4
	Average	535.9	2017.6	1199.9	548.1	17.5	0.588	15.3
	Std Dev	6.2	13.8	52.6	27.1	0.8	0.023	0.3
Sample 3[e]								
1	25	532	2018	1312	530	17	0.574	15.3
2	27	532	2013	1348	489	14	0.491	15.2
3	28	541	2013	1308	508	16	0.546	15.2
4	29	532	2016	1379	511	17	0.574	15.2
5	30	532	2013	1371	508	14	0.491	15.2
6	31	542	2020	1254	504	15	0.518	15.3
7	32	540	2018	1331	512	16	0.546	15.3
8	33	541	2012	1347	484	17	0.574	15.1
9	34	540	2015	1345	464	16	0.546	15.2
10	38	539	2016	1416	520	15	0.518	15.2
11	39	538	2014	1376	507	16	0.546	15.2
12	40	533	2043	1479	540	17	0.574	15.9
	Average	536.9	2017.6	1355.4	506.5	15.8	0.542	15.3
	Std Dev	4.0	8.4	56.8	20.4	1.1	0.031	0.2

[a] Cum Exp = Cumulative Exposure
[b] BF = Back Face
[c] Nextel 440/Saffil blanket with a brush-on PCC coating (areal weight 0.113 lb/ft²)
[d] Nextel 440/Saffil blanket with a spray-on PCC coating (areal weight 0.051 lb/ft²)

Table 1(b). Summary results of arc jet tests on gray and standard C-9 coated Insulations

Cum Exp	Run No.	Time (s) In Arc	Max Surface Temp °F	Time (s) to Max BF	Max BF Temp °F	Chamber Pressure PSIA	Stagnation Pressure PSIA	Calc. Heat Flux Rate Btu/ft^{2}s
Sample 1^a								
1	17	393	2006	1281	443	14	0.491	15.0
2	21	530	1998	1180	537	14	0.491	14.8
3	22	540	1992	1130	510	14	0.491	14.6
4	23	533	2008	1184	541	16	0.546	15.0
5	24	531	2008	1304	541	15	0.518	15.0
6	25	534	2004	1279	534	17	0.574	15.0
	Average	534	2002	1215	533	15	0.524	14.9
	Std Dev	3.9	7.0	73.2	12.9	1.3	0.036	0.2
Sample 2^b								
2^c	45	558	2048	924	604	10	0.379	7.6
3	46	542	2047	920	555	10	0.379	7.5
4	47	541	2062	955	578	9	0.351	7.7
	Average	547	2052	933	579	9.6	0.367	7.6
	Std Dev	11.2	1.2	2.5	34.6	0.0	0.010	0.0

[a] Nextel 440/Saffil blanket with a brush-on gray C-9 coating (areal weight 0.045 lb/ft^2).
[b] Pure Silica AFRSI/Q-felt blanket with a brush-on standard C-9 coating (areal weight 0.158 lb/ft^2).
[c] Sample was uncoated for the first exposure

The gray C-9 coating did not perform quite as well as the PCC, with a hotter average maximum back-face temperature of 530°F at about 1200 seconds elapsed time. In contrast, the standard C-9 yielded the highest back-face temperature of 580°F and the highest thermal conduction rate at 920 seconds elapsed time. This behavior was expected because standard C-9 contains no high emissivity agents for rejection of heat at the surface.

It must be noted the differences observed between the three PCC coated samples and the gray C-9 may be within the limits of the experimental error. The surface coverage can affect the emissivity values and resulting surface temperatures. Also, the placement of thermocouples in the blankets is not precise. If there is any compression of the blankets, the distance between front- and back-face thermocouples may vary and give inconsistent values for thermal conduction rates. It would be reasonable to point out that within the limits of the experimental uncertainties, the PCC and gray C-9 perform equally well under similar arc jet exposure. In order to make exact

measurements of differences in coating thermal performance it would be more desirable to coat a rigid material and do systematic studies on different areal coverages. This was not done here because the main goal of this study was to determine the mechanisms of blanket surface embrittlement.

Impact Testing of Blankets Exposed in the Arc Jet

After the aerothermal exposures in the arc jet, the coated samples were subjected to low energy impact tests in the Ames Materials Testing Laboratory. The test apparatus consisted of a known mass calibrated at three height levels to yield 100, 300, and 500 mJ impact energies. The impacted mass is a 45° (from centerline) conical shape with a nose tip radius of 1/16 inch. Typically, after impact testing on rigid materials, the diameter of the craters created on the surface are measured for comparison. However, on flexible blanket materials, comparison of crater diameter measurements are misleading due to the ability
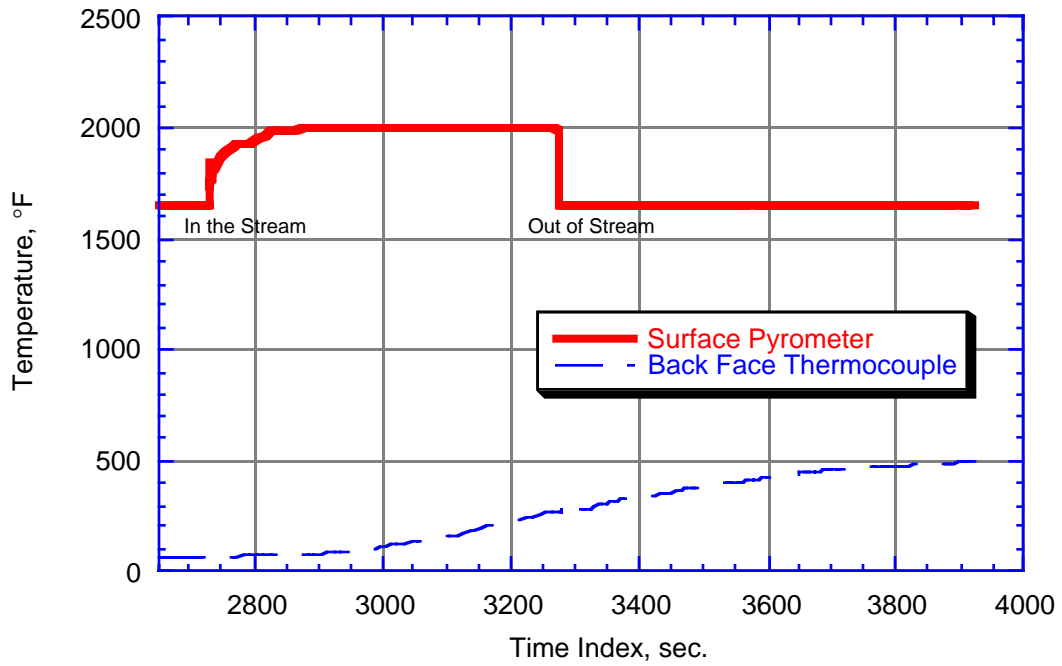
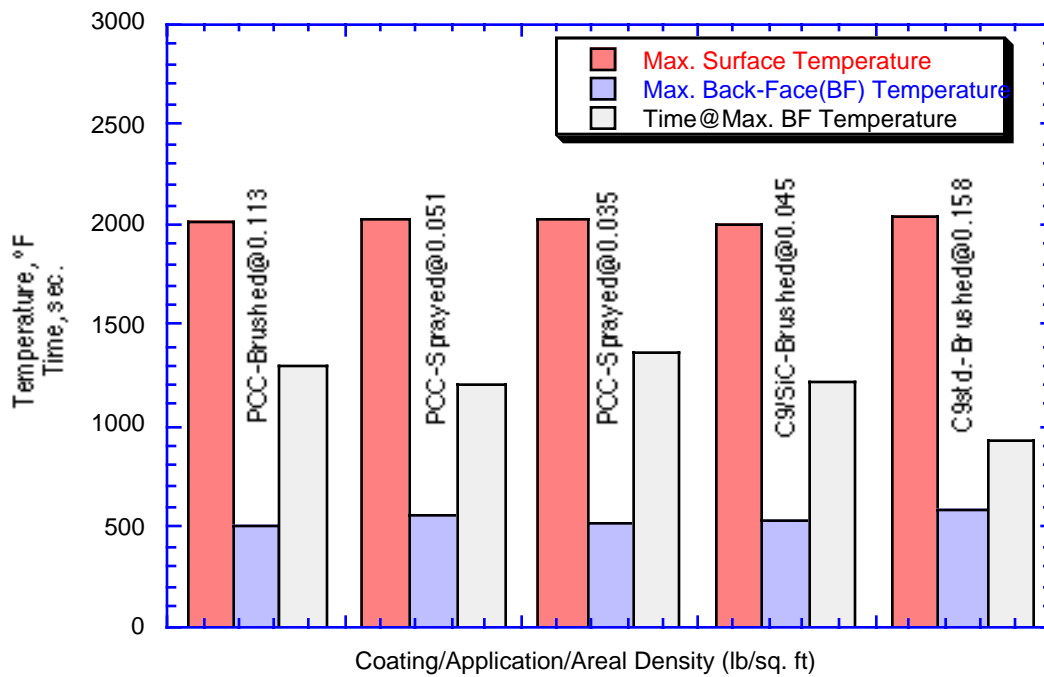*Figure 1. Typical aerothermal cycle of nextel 440 samples.*

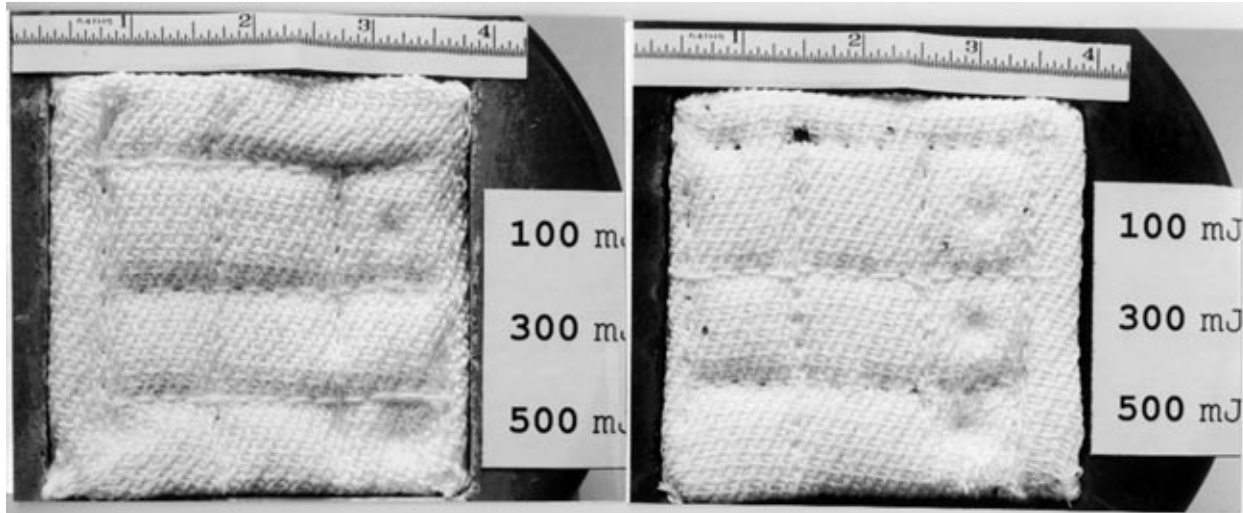*Figure 2. Thermal performance of ceramic coatings.*

Figure 3. Uncoated Nextel 440 AFRSI blankets (postimpact test). (a) 6 exposures in arc jet; (b) 12 exposures in arc jet.

of the surface to flex and return to its original shape. It is more important to make qualitative observations of surface destruction (i.e., cracks in the coating, OML fiber breakage). The results will be discussed in these terms rather than as a quantitative comparison.

Uncoated Blankets

Uncoated Nextel 440 samples, exposed to 6 and 12 arc jet cycles, have a slightly better resistance to impact than coated materials due to the Nextel OML ability to flex and absorb part of the impact energy through dissipation to the surrounding material. Flexing also allows impact energy to be absorbed by the batting instead of the surface fabric. However, with longer exposure to the aerothermal environment in the arc jet, more embrittlement is apparent. This increased embrittlement most likely results from the fibers beginning to sinter and fuse together at high temperature. Thus, it was expected there would be more damage during impact testing to the uncoated sample with 12 heating cycles compared to the one that underwent 6 cycles. Figure 3 shows that the blanket exposed for 6 cycles remained flexible enough so that when the 500 mJ impact took place, the dimple formed during the 300 mJ impact flexed back to its original shape. There was no fiber damage to this blanket due to impact. However, the blanket that was exposed for 12 cycles in the arc jet was brittle enough so that some of the surface fibers and yarns broke during impact at all energies, exposing the internal batting material

Coated Blankets

C-9, Gray C-9 – Figure 4(a) shows the standard shuttle AFRSI coated with standard C-9 (brush-on application, areal density of 0.158 lb/ft^2) that has gone through four exposures in the arc jet and subsequent impact testing. Note that standard shuttle AFRSI is composed of a silica fabric OML and a Q-felt batting. This pure silica blanket is made for lower temperature use than the Nextel 440 with Saffil batting. The blanket is extremely rigid after only four cycles in the arc jet. Impact testing did serious damage, totally exposing the batting material to a depth of ~1/4 inch. The damage is likely due to a combination of the relatively high areal density of the coating as well as significant embrittlement of the coating, the OML, and batting material.

Figure 4(b) shows a Nextel 440/Saffil blanket coated with gray C-9 (brush on application, 0.045 lb/ft^2) that has been through six cycles in the arc jet and then impact-tested. It is barely evident that impact testing has been carried out on this sample. Some of the coating was removed during impact but no damage to fibers is observed. This represents good application of the coating although there is not full surface coverage. Note the small holes in the coating where hot gas can flow to the interior of the material during arc jet testing. This may be partially responsible for the back-face temperatures being slightly higher for this sample than for the PCC coated materials. Application of a higher areal density gray C-9 coating will most likely result in greater impact damage. More work will be carried out to optimize the coating areal density and application procedure.
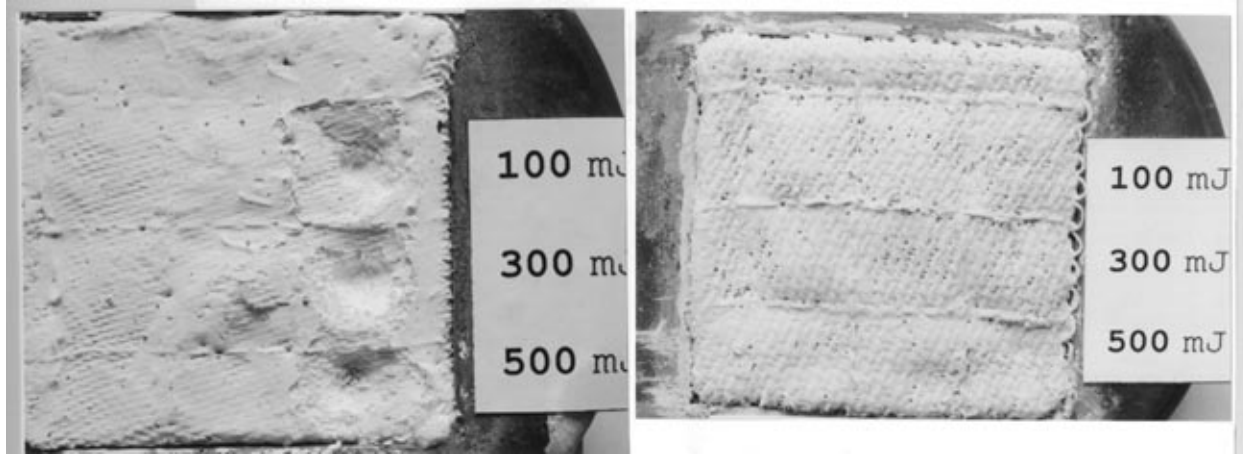
Figure 4. C-9 coatings on AFRSI (postimpact test). (a) Standard C-9 coating (0.158 lb/ft^2) on standard (pure silica) AFRSI; (b) gray C-9 (0.045 lb/ft^2) on Nextel 440/Saffil AFRSI.
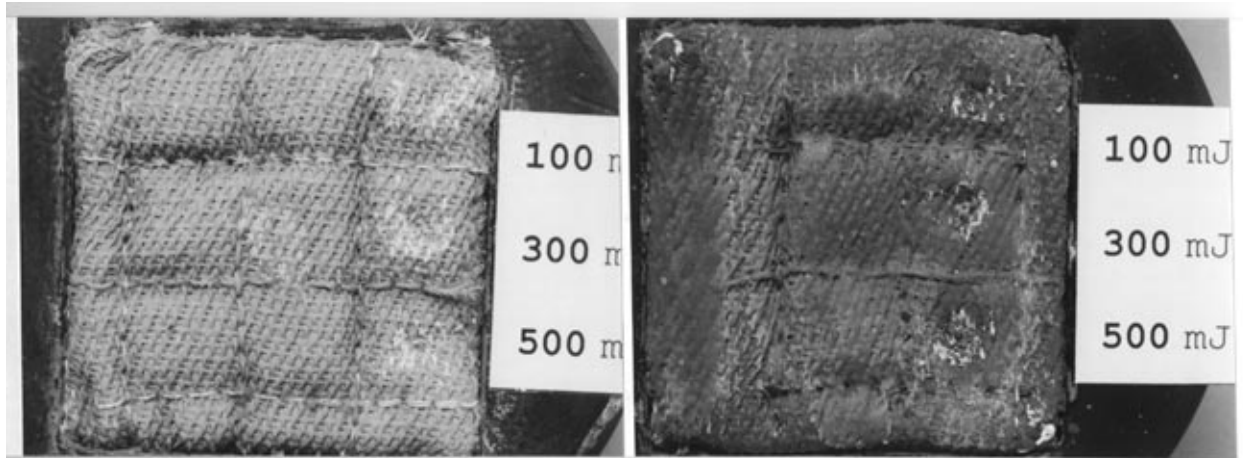

Figure 5. PCC coatings on Nextel 440/Saffil AFRSI (postimpact test). (a) Spray-on (0.051 lb/ft^2); (b) brush-on (0.113 lb/ft^2).

PCC – Figure 5 compares two Nextel 440/Saffil blankets coated by different application methods of PCC. Both materials were exposed in the arc jet for six cycles. The sample on the left was coated by a spray on application with a low areal density of 0.051 lb/ft^2. The coating on the right was brushed on with a relatively high areal coverage of 0.113 lb/ft^2. Impact testing on the lower density coating gave results similar to those observed for gray C-9. Some of the coating was removed during impact but no fiber damage was observed. The removal of coating is more evident in this photo than in the gray C-9 photo because of the darker shade of PCC.

There was a great deal of damage to high areal density PCC coated blanket. The resulting craters were about 1/8 inch deep, and quite a bit of OML fiber damage is apparent although not enough to remove the fabric and expose batting material. It was initially speculated that the SiB$_6$ in the PCC coating rigidized the Nextel yarns because the boron reacted with the 2% B$_2$O$_3$ of the virgin Nextel material. However, a wavelength dispersive x-ray analysis of the fiber crossections indicates that there is no chemical reaction between the fibers and the coating. The boron concentrations throughout the fibers are the same before and after exposure to the arc jet. It is more likely that thicker coatings lead to mechanical failure rather than any chemical reactions of the emissivity agents with the

8

OML. SEM analyses of coated fabric cross sections were obtained from pre and post arc jet tested samples. The posttest PCC, gray C-9, and standard C-9 coatings all appear smoother than the pretest samples. The glassy texture indicates that some degree of sintering or melting has occurred during high temperature cycling. The process binds the OML yarns to some extent (depending on the initial areal coverage) to a glassy matrix and rigidizes the overall surface of the fabric. Even if the fibers remain flexible after heat treatment, they are unable to flex if encapsulated in glass. When the glass coating fractures, the fibers also fracture. In addition, there is bonding or fusing of the individual fibers within the yarns which further reduces surface flexibility.

Sintering of fibers and/or melting of the coating material is expected in all silica based materials. Increasing the exposure to high temperature cycling will increase these effects. One development goal for these materials is to minimize the degree to which these processes affect the flexibility of the blanket. It is fairly clear from this work that lowering the areal density and thickness of the coating reduces the ability of the coating to harden the surface.

High Temperature Emissivity

The purpose of this work is to provide estimates of the temperature-dependent emissivites of all of the coated blanket samples that were exposed in the arc jet. These emittance estimates are obtained from room temperature hemispherical reflectance measurements using an averaging procedure described below. Estimates are provided from room temperature to 2900 K (2960°F).

Additional coated fabric samples were prepared in order to determine whether the emissivity is different for pretest samples. Nine samples were prepared by coating angle-interlocked Nextel 440 fabric. Four samples were coated with PCC and four with gray C-9; one sample was left uncoated. Each set of four samples was composed of two specimens with sprayed-on coatings and two specimens with brushed-on coatings. Half of these specimens were fired at 2000°F for 1 hour and the other half were left unfired.

The hemispherical spectral reflectance was measured at room temperature over a wavelength range of 0.25 to 22.0 μm. A Perkin-Elmer Lambda-9 spectrophotometer was used for measurements from 0.25 to 2.5 μm and a BIORAD FTS-40 spectrophotometer was used for measurements from 2.5 to 22.0 μm. However, data above 18.0 μm is of questionable accuracy and has been excluded. Most spectra exhibit noise in the region of 2 to 4 μm; however, the average reflectance values in this wave-

length region are believed to be accurate and the noise has little impact on the emittance computations that follow.

Measured hemispherical spectral reflectances ρ are converted to hemispherical spectral emittances ε using the expression

$$\varepsilon(\lambda, T_R) = 1 - \rho(\lambda, T_R)$$

which is valid for a diffusely irradiated opaque surface in thermal equilibrium with its surroundings at temperature T_R. Emittance temperature dependence is estimated by averaging the room temperature spectral emittance values over the Planck distribution function at temperature T, i.e.,

$$\varepsilon(T) = \frac{\int\limits_{\lambda_l}^{\lambda_u} \varepsilon(\lambda, T_R) e_{\lambda b}(\lambda, T) d\lambda}{\int\limits_{\lambda_l}^{\lambda_u} e_{\lambda b}(\lambda, T) d\lambda}$$

Here λ_l and λ_u are, respectively, the lower and upper limits of the wavelength range over which $\rho(\lambda, T_R)$ was measured.

The Planck distribution is given by

$$e_{\lambda b}(\lambda, T) = \frac{2\pi C_1}{\lambda^5 \left(\exp\left\{ \frac{C_2}{\lambda T} \right\} - 1 \right)}$$

with the radiation constants $C_1 = 0.595522e+8$ W-μm^4/m^2-sr and $C_2 = 0.0143877$ μm-K.

Figure 6 shows the fraction of blackbody emissive power which lies in the spectral range of the experimental reflectance measurements at different temperatures. The estimation procedure becomes "better" when this fraction approaches 1. For temperatures above ~525 K this fraction is at least 0.9. However, the estimation procedure employed here assumes that the spectral reflectances measured at room temperature do not have significant temperature dependencies. This may lead to some (unquantified) overestimation of the emittance values at elevated temperatures.

Note that the reflectance based emittance values are in good agreement with elevated temperature emittances obtained using a two-color pyrometer during arc jet testing. This gives independent support of the values presented here. In situ emissivity measurements were made at 2000°F during the first cycle of an arc jet test series on PCC coatings. The PCC coated material (0.074 lb/ft^2) showed an emissivity of ~0.85 and the emissivity estimated from the room temperature measurement of the

posttest sample was ~0.87 at 2000°F. It appears that the extrapolation method used here is a fairly good approximation.

The emittances for PCC (0.051 lb/ft^2), standard C-9 (0.158 lb/ft^2) and a "gray" C-9 coated sample (0.045 lb/ft^2), as well as uncoated Nextel 440 sample are shown in Figure 7. All of the samples were exposed for six cycles in the arc jet except for the standard C-9 which underwent only four exposures. The emittance of the standard C-9 coating on the pure silica AFRSI is substantially lower at elevated temperatures than that of the "gray" C-9 coated Nextel sample, as expected. The standard C-9 gave the highest surface and back face temperatures of any of the coated materials tested. The high

temperature emittance data for the nine samples coated and fired in a furnace are shown in Figures 8 and 9.

The data are consistent with the data obtained from the arc jet exposed materials. The emittance is seen to decrease with increasing temperature in all cases. There does not seem to be any systematic dependence of emittance values on the coating application technique (i.e., sprayed versus brushed). Firing for 60 minutes at 2000°F appears to have lowered the emittance values for the gray C-9 coating but raised them for the PCC coating. There is larger deviation of emittance values among the PCC samples than the gray C-9 values.
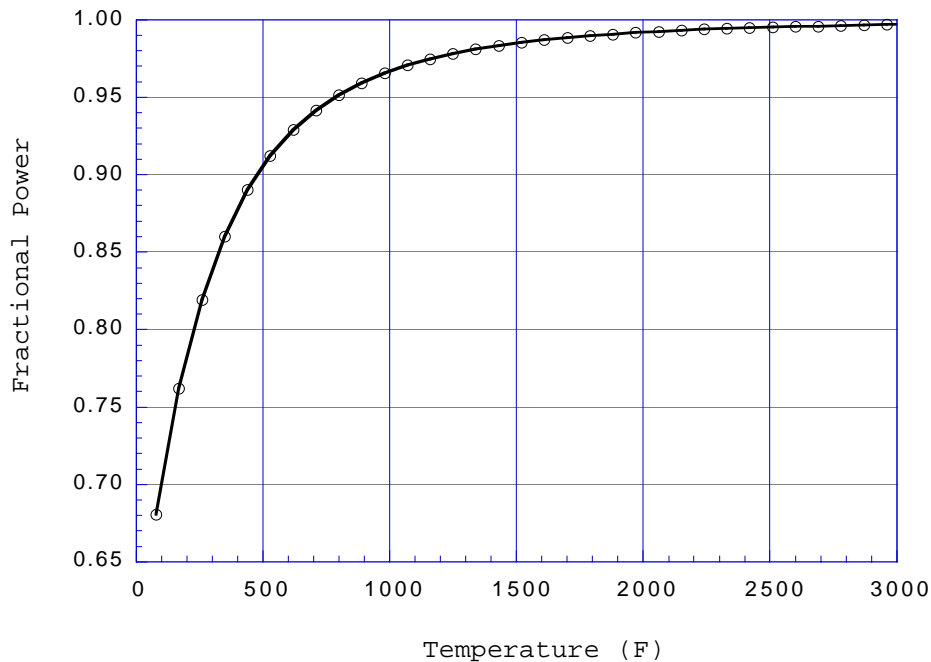

Figure 6. Fraction of blackbody emissive power: Spectral range 0.25 µm to 18.0 µm.
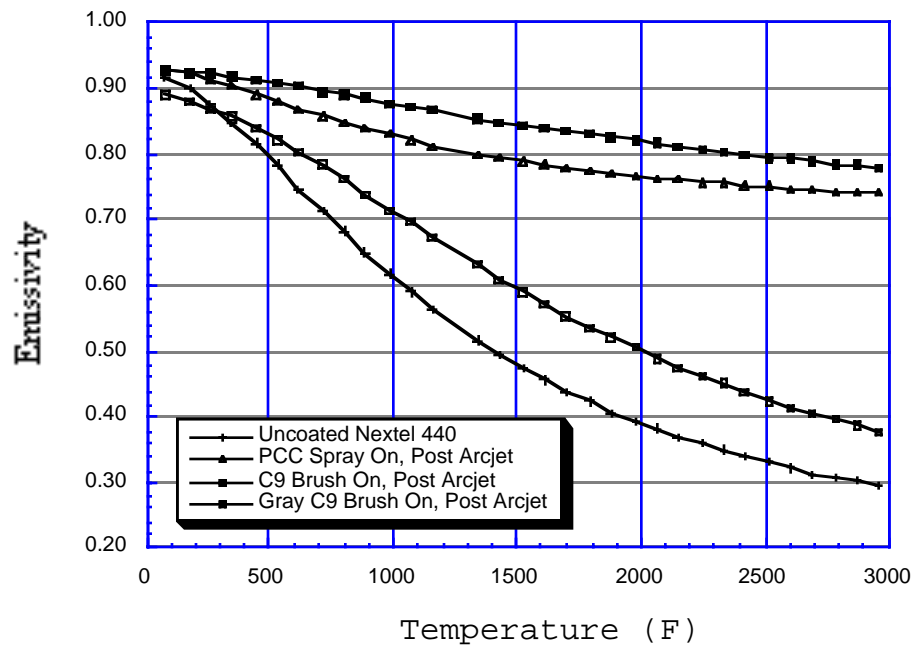
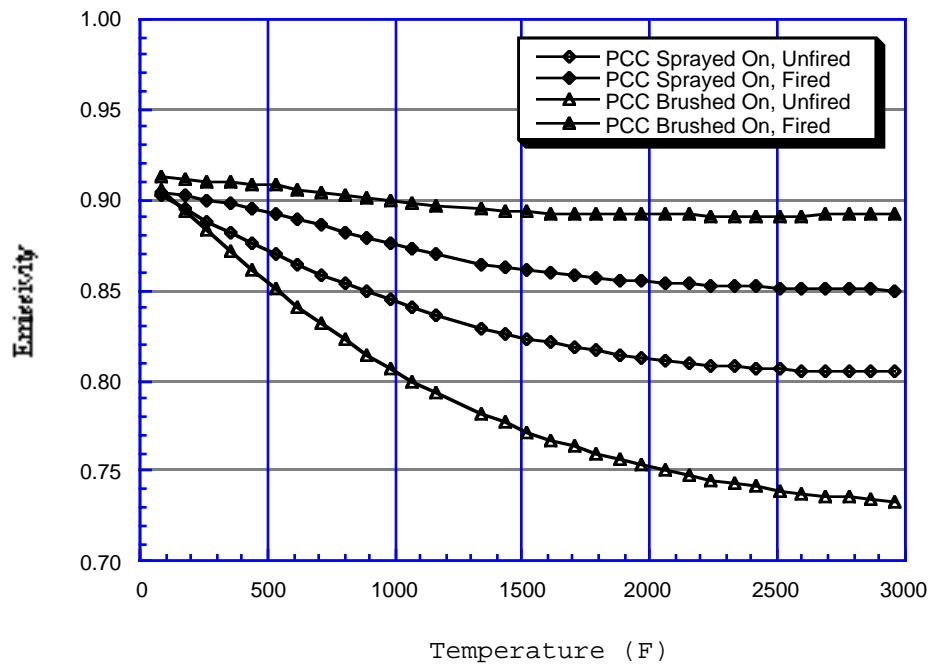Figure 7. Emissivities of post arc jet samples.

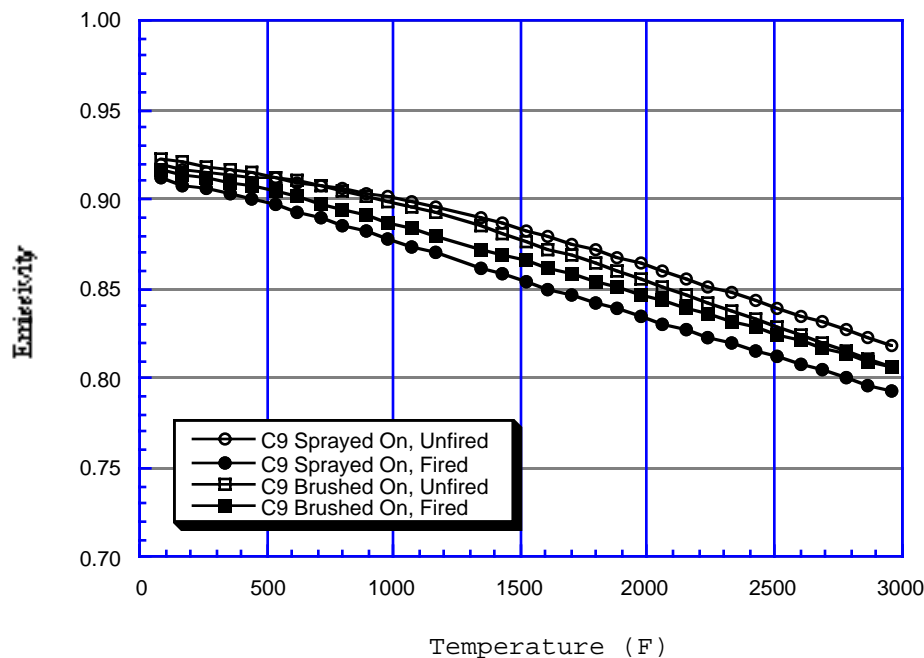Figure 8. Emissivities of PCC coated Nextel 440 fabric.

11

Figure 9. Emissivity of gray C-9 coatings on Nextel 440 fabric.

Conclusions and Future Work

Arc jet testing of PCC and gray C-9 coated Nextel 440/Saffil blankets shows that both coatings meet the goals of the program. These goals include increasing the surface emissivity and reduction of convection penetration of "hot" gas by reducing the OML fabric porosity. Closing the surface pores will also reduce surface catalytic effects. The PCC coating performed slightly better in the arc jet than the gray C-9, presumably due to the higher emissivity for that particular coating formulation. Back-face temperatures were significantly lower for PCC and gray C-9 coated Nextel 440/Saffil blankets than for a standard shuttle blanket with standard C-9 coating.

It is clear from impact testing that any of these silica based surface coatings will embrittle flexible ceramic materials to some degree when exposed to high temperatures. The coatings reduce the ability of the Nextel 440 fabric to flex, to absorb impact energy, and to transmit energy to the batting—but not severely for samples with lower surface coverage. There are three mechanisms involved in the surface embrittlement process. First, the OML fibers themselves can begin to sinter at high temperatures and the amorphous material will begin to

crystallize, which causes rigidization of the individual fibers. The sintering process will also cause fusing of adjacent fibers, which rigidizes the entire OML fabric.[2] In addition, the high emissivity coatings applied to the surface will rigidize when exposed to high temperature. This hard coating encases the yarns and fibers of the OML fusing them together into one solid system, which causes further embrittlement. This embrittlement factor is clearly dependent on the amount of coating applied. Low areal density coating applications reduce the number of yarns and fibers encased by the hard coating and subsequently lessen the degree of fusing of the individual fibers and strands of OML yarn at the upper use temperature of the coating/fabric.

In future work, the elevated temperature emittances of these coatings will be measured at actual temperature, using a controlled experimental apparatus along the lines described in reference 4. Such an apparatus consists of a tube furnace and temperature controller, a sight tube and

---

[2] Determining which of these two processes is more prevalent requires further study. The fusing of fibers has been observed in SEM images, but to observe the crystallization process it will be necessary to measure x-ray diffraction patterns of fibers before and after a series of heat treatments.

sight tube positioning mechanism, a radiometer, a radiometric zero, a data acquisition system, and an integral blackbody and test specimen fixture. Use of an integral blackbody and test specimen fixture assures that both the sample and the blackbody reference are at the same temperature during a test. This fixture is placed inside the furnace with the sample normal to the tube axis. When viewed by the radiometer along the tube axis this configuration acts as a cylindrical cavity blackbody, and when the sight tube is inserted only radiation emitted by the sample is viewed. The ratio of radiometer voltages under these two conditions gives the emittance. An apparatus similiar to this is being designed, and the necessary tests will be carried out in future work.

Several other experiments will also be carried out. The effect of firing and arc jet exposure on emittance is not yet well characterized because sample-to-sample coating variation may interfere with intersample comparisons. A simple experiment would be to perform reflectance measurements on the same samples before and after firing or arc jet exposure. Additionally, it would be informative to determine the minimum coating thicknesses necessary to assure that the surface emittance is determined entirely by the coating. Dielectrics emit radiation from a near surface volume of material; if the coating is too thin, the surface emittance will be influenced by the fabric substrate. A straightforward experiment would be to map changes in the reflectance spectra with increasing coating thickness.

References

1. Nextel 440 Ceramic Fiber. 3M Ceramic Fiber Products Technical Bulletin.

2. Kourtides, D; Churchward, R; and Lowe, D: Protective Coating for Ceramic Materials. United States Government patent #5296288, March 22, 1994.

3. Mui, D.; and Clancy, H. M.: Development of a Protective Ceramic Coating for Shuttle Orbiter Advanced Flexible Reusable Surface Insulation (AFRSI). Ceramic Engineering and Science Proceedings, vol. 6, no. 7-8, Jul.-Aug. 1985, pp. 793-805

4. Vader, Viskanta and Incropera, Rev. Sci. Instrum., vol. 57, no. 87, 1986.

REPORT DOCUMENTATION PAGE

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	July 1997	Technical Memorandum

4. TITLE AND SUBTITLE

Evaluation of Thermal Control Coatings for Flexible Ceramic Thermal Protection Systems

6. AUTHOR(S)

Demetrius Kourtides, Carol Carroll, Mike Guzinski,* Jochen Marschall,* Joan Pallix,* Jerry Ridge,* Dane Smith, and Duoc Tran*

5. FUNDING NUMBERS

242-20-01

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Ames Research Center
Moffett Field, CA 94035-1000

8. PERFORMING ORGANIZATION REPORT NUMBER

A-976757

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, DC 20546-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NASA TM-112199

11. SUPPLEMENTARY NOTES

Point of Contact: Dane Smith, Ames Research Center, MS 234-1, Moffett Field, CA 94035-1000;
(415) 604-6080
*Thermosciences Institute, Moffett Field, CA 94035-1000

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified — Unlimited
Subject Category 27

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

This report summarizes the evaluation and testing of high emissivity protective coatings applied to flexible insulations for the Reusable Launch Vehicle technology program. Ceramic coatings were evaluated for their thermal properties, durability, and potential for reuse. One of the major goals was to determine the mechanism by which these coated blanket surfaces become brittle and try to modify the coatings to reduce or eliminate embrittlement. Coatings were prepared from colloidal silica with a small percentage of either SiC or SiB_6 as the emissivity agent. These coatings are referred to as gray C-9[1] and protective ceramic coating (PCC), respectively. The colloidal solutions were either brushed or sprayed onto advanced flexible reusable surface insulation blankets. The blankets were instrumented with thermocouples and exposed to reentry heating conditions in the Ames Aeroheating Arc Jet Facility. Posttest samples were then characterized through impact testing, emissivity measurements, chemical analysis, and observation of changes in surface morphology. The results show that both coatings performed well in arc jet tests with backface temperatures slightly lower for the PCC coating than with gray C-9. Impact testing showed that the least extensive surface destruction was experienced on blankets with lower areal density coatings.

14. SUBJECT TERMS

Thermal protection systems, Thermal control coatings, emissivities

15. NUMBER OF PAGES

15

16. PRICE CODE

A03

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified		